doi: 10.1017/S1431927609097529
Microsc Microanal 15(Suppl 2), 2009

SEM of Carbon Coated $LiFePO_4$ Through Silicon Nitride Windows in Liquid

Jane Y. Howe, Larry R. Walker, Jim O. Kiggans, and Nancy J. Dudney

Materials Science & Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831

This paper reports a scanning electron microscopy (SEM) initial examination of Li-ion battery materials through a thin silicon nitride window. Although "wet" SEM using a Quantomix cell has been achieved, the Quantomix cell's polymeric window prohibits any heating experiments [1]. In contrast, amorphous silicon nitride windows, such as these produced by Protochips Inc. can be heated up to 1000 ^{o}C within seconds and endure hours of heat treatment. Proven durability of these windows in transmission electron microscopy applications [2] inspired us to explore the possibility of constructing a liquid flow cell for *in situ* SEM studies. A set of proof-of-principle experiments have been carried out using a Hitachi S3400 Variable Pressure SEM at 5, 7, 10, 15, 20, and 25 kV accelerating voltages. EDS spectra and elemental maps were undertaken using an EDAX Si(Li) spectrometer. We used silicon nitride windows of 50, 100, and 200 nm in thickness: a thin silicon nitride film is coated on a silicon chip with a 0.5 x 0.5 mm^2 opening. A paste consisting of $LiFePO_4$ powder and AR mesophase pitch in N-vinyl-2-pyrrolidone suspension was used for the study. A droplet of the paste was dispended into the dent of the chip, then heated to 700 ^{o}C in flowing argon for 5 h in order to carbonize the pitch. The chips were then placed on an aluminum stub for imaging and EDS analysis. Chips with 100 and 200 nm windows withstood the heat treatment. Fig. 1 is a backscattered micrograph along with element maps using a 5 kV electron beam through a 100 nm window. Through-window-images show some resolution loss and particles quickly lose focus situated away from the window. After initial imaging, a droplet of ethylene glycol was added to the cavity and then sealed. Images of particles fully submerged in liquid are presented in Fig. 2. EDS analysis has been undertaken on ethylene glycol as well as particles submerged in the liquid. Charging in the non-polar liquid was a problem at lower voltages but alleviated at accelerating voltage of 25 kV.

In summary, SEM study has been carried out on carbon coated $LiFePO_4$ powders through a 100-nm silicon nitride window at resolution of 100 nm or better. Elemental mapping can be achieved at accelerating voltages as low as 5 kV without the presence of liquid. A comparison study using Quantomix cells is also presented. [3]

References

[1] S. Thiberge, O. Zik, E. Moses, Rev. Sci. Inst. 75(7), 2280-2289 (2004).
[2] N. De Jonge, D.B. Peckys, G.J. Kremers, et al, Proc. Nat. Aca. Sci. Online Ed. Jan. 21, 2009.
[3] Research at the ORNL through the High Temperature Materials Laboratory was sponsored by the Assistant Secretary for Energy-Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC. We thank Protochips, Inc. for supplies and insightful discussions.

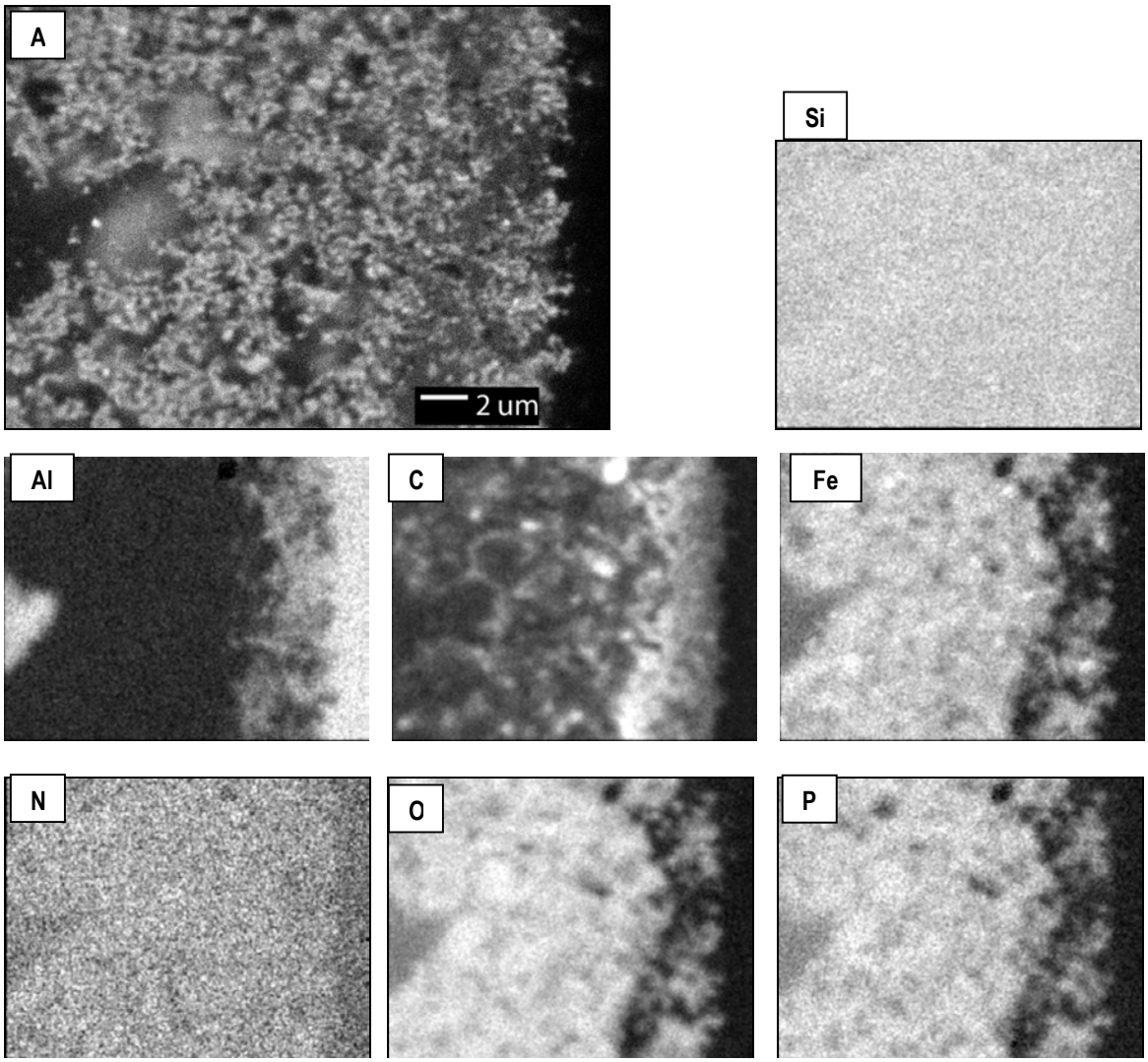

Fig. 1. BSE image (a) and element maps of $LiFePO_4/C$ through the 100-nm silicon nitride window.

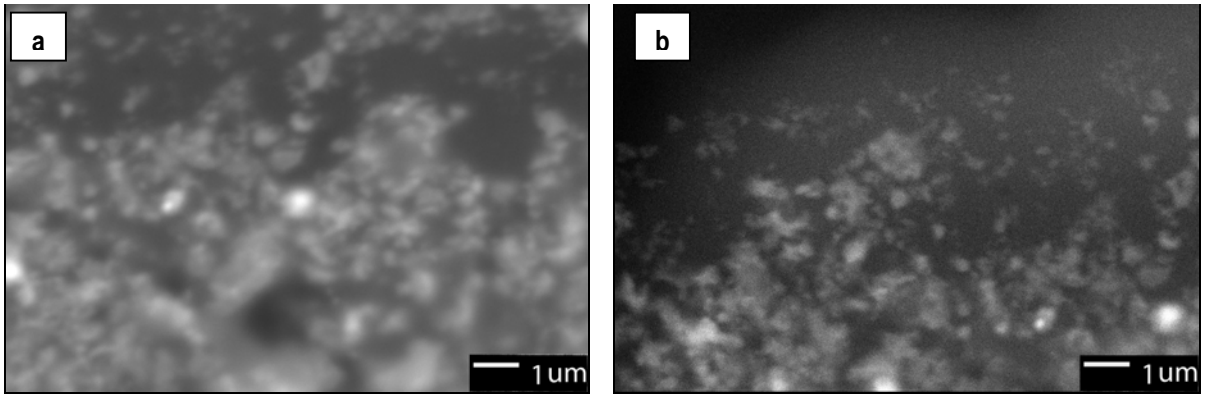

Fig. 2. Micrographs of $LiFePO_4/C$ in ethylene glycol through the window at 15 (a) and 25 kV (b).

https://doi.org/10.1017/S1431927609097529 Published online by Cambridge University Press